

# SCIENCE FOR CERAMIC PRODUCTION

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## PRODUCTION OF REINFORCED CERAMICS BASED ON ALUMINUM NITRIDE

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The mechanism of the formation of aluminum nitride ceramics with a fibrous structure is considered. The possibility of isomorphic substitution of Si, Mg, Ca, Y, and other more available rare-earth elements in the aluminum lattice is demonstrated.

Ceramics reinforced with fibers possess mechanical properties which are different from the properties of ceramics with a polydisperse structure [1]. Fiber-reinforced ceramics acquire plasticity connected with the mechanism of “pulling fibers out of the matrix” [2]. Figure 1 shows a diagram of the destruction of ceramic material in the course of crack growth.

It is important to comprehend the formation mechanism of fibrous-structure ceramics. It is established in [3] that  $\text{SiO}_2$  additive facilitates the formation of a new ceramic microstructure. In the presence of  $\text{SiO}_2$ , the polytype 27R with plate-shaped grains emerges. Whereas its introduction in pure form impedes condensation, when it is added together with yttrium oxide (~3 wt.%), a high-density ceramic material with good mechanical properties is formed [3]. As the silicon oxide content increases, the new composition – Al–Si–O–N– is formed, whose parameters are somewhat modified compared to the aluminum nitride lattice: the parameter  $a$  is shortened, and the parameter  $c$  is lengthened. Materials in this system were named “sialons.” The system manifests the polytypism of aluminum nitride. Table 1 presents AlN polytypes formed in the Al–Si–O–N system [4].

It can be assumed that two processes take place in sintering of ceramics based on aluminum nitride: isomorphic atom substitution in the crystal lattice and interphase interaction at the phase boundary in the course of grain formation. The correlation of these two processes determines the possibility of structure formation.

Since the emerging systems contain a great amount of phases and compounds, the study is carried out using the

method of correlation of properties and structures in modifying the initial-component composition or the sintering technology. Atoms of Si, Mg, Ca, Y, and other rare-earth ele-

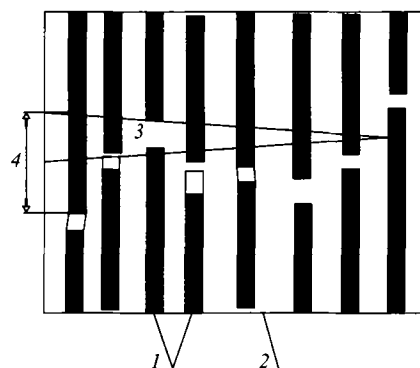


Fig. 1. Scheme of pulling out a fiber from brittle ceramic matrix: 1) fiber; 2) matrix; 3) crack in the matrix; 4) pulled part of the fiber.

TABLE 1

Phase index	Cell parameters		
	$a$ , Å	$c$ , Å	M/NM*
8H	2.988	23.02	4/5
15R	3.010	41.81	5/6
12H	3.029	32.91	6/7
21R	3.048	57.19	7/8
21R	3.059	71.98	8/9
2H	3.079	5.30	9/10

\* M) number of metal atoms; NM) number of nonmetal atoms per elementary cell.

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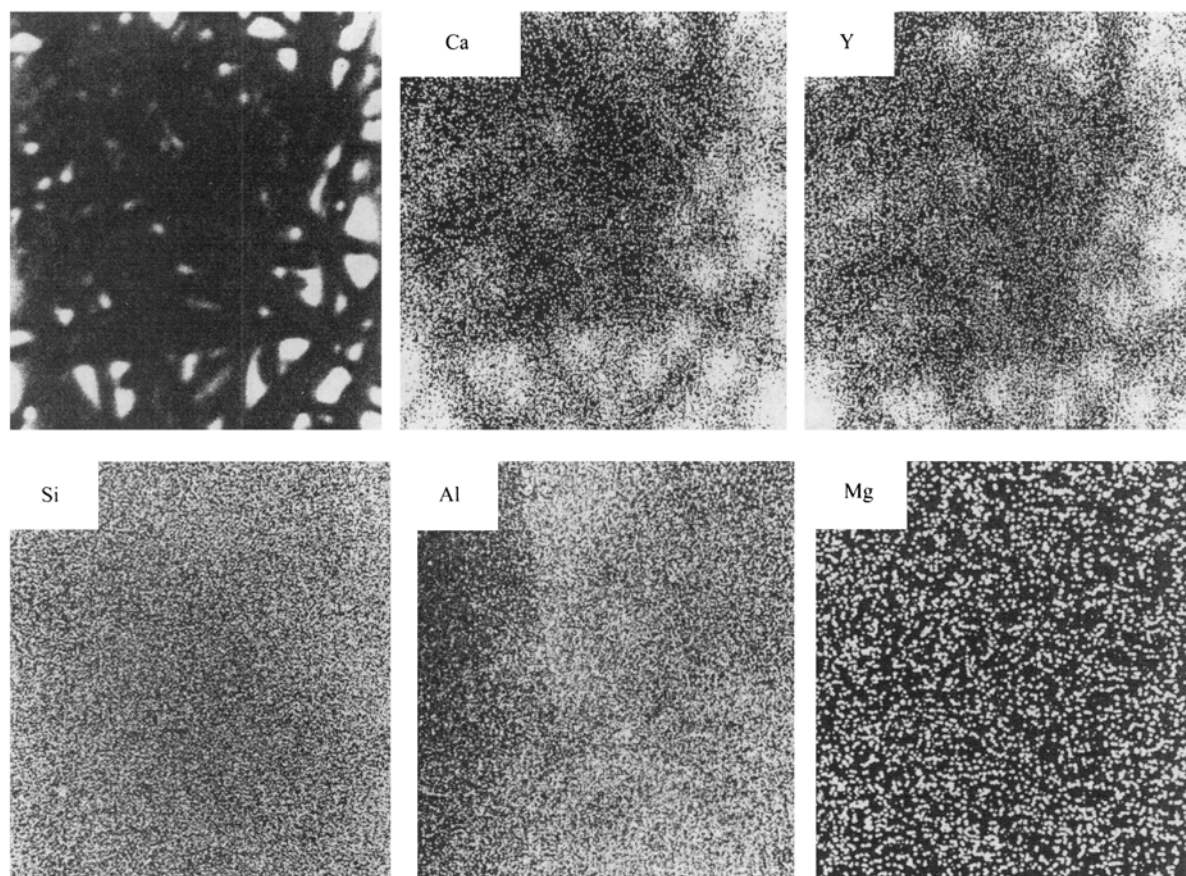


Fig. 2. Ceramics structure in reflected electrons and characteristic x-rays.

ments are considered as atoms capable of isomorphic substitution in the aluminum lattice. To determine the effect of these elements on the structure formation, additives were chosen in such a way as to form a series based on atom sizes. The atoms introduced into ceramics in sintering and their crystallographic radii were as follows (nm): Mg — 0.160, Ca — 0.197, Al — 0.143, Si — 0.134, Y — 0.181, Pr — 0.182, Dy — 0.177, N — 0.148, and O — 0.136 [5].

An efficient technology modifying the structure and properties of a disperse powder consists of the variation of the initial powder dispersion, which makes it possible to form a finely crystalline structure, increase the sintering rate, and presumably facilitate the formation of intermediate phases.

In order to determine the effect of the dispersion degree of the sintering additive on ceramic structure and properties, sintering of aluminum nitride was investigated. The yttrium oxide sintering additive in an ultradisperse state ( $\sim 0.01 \mu\text{m}$ ) and aluminum nitride (dispersion  $1 - 10 \mu\text{m}$ ) were mixed and then sintered according to the method described in [1]. Figure 2 shows a microphoto of the structural ceramics based on AlN. The sintering additive was 5 wt.%  $\text{Y}_2\text{O}_3$  in the form of ultradisperse powder produced by the plasma-chemical

method, and silicon, magnesium and calcium additives were added as well.

The main structural element of the obtained ceramics is fiber. It can be seen in the photo of the polished ceramic section that its minimum porosity can be 1 – 2%. The structure of the ceramics consists of agglomerated needles and plates of aluminum nitride with the aluminum-yttrium garnet phase  $\text{Y}_4\text{Al}_2\text{O}_9$  located between the plates. The oxide phase is

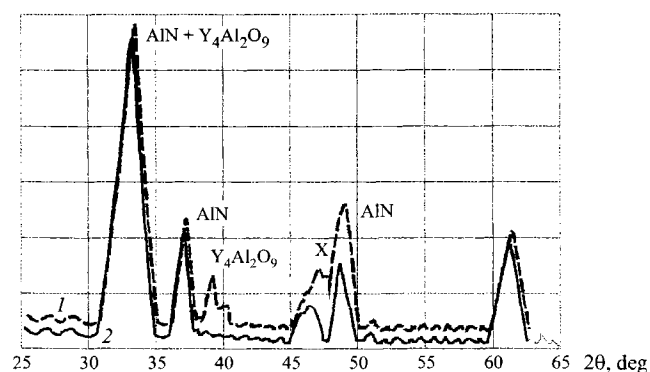


Fig. 3. X-ray patterns of ceramics produced in the AlN –  $\text{Y}_2\text{O}_3$  system with additives (1) and according to Komeya's data [3] (2).

probably located between the fibers. The photos obtained in characteristic x-rays show that Ca and Y follow the fibrous structure of ceramics, whereas Si, Al, and Mg are virtually uniformly distributed over the entire area of the photo (Fig. 2).

The atomic sizes of Ca and Y are slightly greater than the size of Si, Al, and Mg. However, three atoms, i.e., Si, Al, and O, constitute the aluminum nitride structure. Judging from the size of the Mg atoms, it can be assumed that Mg is capable of replacing Al in aluminum nitride or existing at the interphase boundary. It is established that silicon, oxygen, and aluminum can be part of the aluminum nitride structure, and yet in the photos they do not follow the fibrous structure, and neither does Mg. The x-ray phase analysis (Fig. 3) shows that the ceramic contains aluminum-yttrium garnet, which is located at the interface with aluminum nitride. Therefore, it can be assumed that Ca and Y are arranged not inside the AlN phase, i.e., they do not replace AlN atoms but form a separate phase, and these separate phases form a fibrous structure, possibly following the "vapor – liquid – crystal" mechanism [1].

Owing to pulverization of  $Y_2O_3$ , the conditions of its reaction with  $Al_2O_3$  located on the surface of AlN grains were modified. The emerging  $Y_4Al_2O_9$  probably reveals its surfactant properties, which determine the directed growth of the AlN crystal. A modification in the dispersion degree of one ingredient significantly modified the ceramic material structure. On the whole, the system did not change, and only the terms of the sintering-additive formation were changed.

The disadvantage of the type of ceramics considered lies in the fact that despite the obvious simplification of the sintering technology for reinforced ceramics, it involves such a scarce and expensive material as yttrium oxide.

That is why a sintering additive consisting of rare-earth oxides with a partial content of calcium and silicon oxides is of great significance. The research results show that rare-earth element oxides behave in the same way as yttrium oxide. It is established that praseodymium and dysprosium oxides, the same as Y and Ca, conform to the fibrous ceramic structure.

Thus, the formation of fibrous-structured ceramics determines the presence of a phase located on the surface of aluminum nitride. Furthermore, one of the ingredients of the sintering additive should be in a highly disperse state.

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